PB89-237382

Performance Audit Results for Volatile POHC (Principal Organic Hazardous Constituents) Measurements during RCRA (Resource Conservation and Recovery Act) Trial Burn Tests

Research Triangle Inst., Research Triangle Park, NC

Prepared for:

Environmental Monitoring Systems Lab., Research Triangle Park, NC

Jun 88

U.S. Department of Commerce
National Technical Information Service
NTIS

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO.	2.	3. PB89-237382
EPA/600/J-88/363		

4. TITLE AND SUBTITLE	5. REPORT DATE
Performance Audit Results for Volatile Principal Organic Hazardous Constituent (POHC) Measurements During RCRA Trial Burn Tests	March 1988
	6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S)	8. PERFORMING ORGANIZATION REPORT NO.
R.K.M.Jayanty, C. K. Sokol, and D. J. von Lehmden	
9. PERFORMING ORGANIZATION NAME AND ADDRESS	10. PROGRAM ELEMENT NO.
Research Triangle Institute P. O. Box 12194 Research Triangle Park, NC 27709	HW - Project Q0101
	11. CONTRACT/GRANT NO.
12. SPONSORING AGENCY NAME AND ADDRESS	13. TYPE OF REPORT AND PERIOD COVERED
U.S. Environmental Protection Agency Office of Research and Development Environmental Monitoring Systems Laboratory Research Triangle Park, NC 27711	14. SPONSORING AGENCY CODE

15. SUPPLEMENTARY NOTES

16. ABSTRACT

Audit materials containing Principal Organic Hazardous Constituents (POHCs) have been developed by Environmental Monitoring Systems Laboratory (RTP) for use by federal state, and local agencies or their contractors to assess the accuracy of measurement methods used during RCRA trial burn tests. Audit materials are currently available for 27 gaseous organics in five, six, seven, and nine component mixtures at parts-per-billion levels (7 to 10,000 ppb) in compressed gas cylinders in a balance gas of nitrogen. Stability studies indicate that all of the organics tested (with the exception of ethylene oxide and propylene oxide below 10 ppb levels) are stable enough to be used as reliable audit materials.

Subsequent to completion of the stability studies, 89 performance audits have been conducted with the audit materials to assess the accuracy of the Volatile Organic Sampling Train (VOST) and bag measurement methods during or prior to RCRA trial burn tests. A summary of the audits conducted for each POHC and the measurement system audited is shown in a technical paper accepted for publicatin by JAPCA. The audit results obtained with audit gases during RCRA trial burn tests are generally within ± 50 percent of the audit concentrations.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Principal Organic Hazardous Constituents (POHC), RCRA Trial Burn Tests, Volatile Organic Sampling Train (VOST), Performance Audits		

REPRODUCED BY
U.S. DEPARTMENT OF COMMERCE
NATIONAL TECHNICAL INFORMATION SERVICE
SPRINGFIELD, VA. 22161

18. DISTRIBUTION STATEMENT	19. SECURITY CLASS (This Report)	21. NO. OF PAGES
Release to public	Unclassified	7
	20. SECURITY CLASS (This page)	22. PRICE
	Unclassified	

EPA/600/J-88/363

Reprinted from APCA Journal, Vol. 38, No. 6, July 1988

Performance Audit Results for Volatile POHC Measurements

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Audit materials containing principal organic hazardous constituents (POHCs) have been developed by EPA for use by federal, state, and local agencies or their contractors to assess the accuracy of measurement methods used during RCRA trial burn tests. Audit materials are currently available for 27 gaseous organics in five, six, seven, and nine-component mixtures at parts-per-billion levels (7 to 10,000 ppb) in compressed gas cylinders in a balance gas of nitrogen. The criteria used for the selection of 27 gaseous organic compounds is described.

Stability studies indicate that all of the organics tested (with the exception of ethylene oxide and propylene oxide below 10 ppb levels) are stable enough to be used as reliable audit materials.

Subsequent to completion of the stability studies, 89 performance audits have been conducted with the audit materials to assess the accuracy of the Volatile Organic Sampling Train (VOST) and bag measurement methods during or prior to RCRA trial burn tests. A summary of the audits conducted for each POHC and the measurement system audited is shown in this paper. The audit results obtained with audit gases during RCRA trial burn tests are generally within ±50 percent of the audit concentrations.

The determination of POHC at trace levels (<50 ppb levels) during hazardous waste trial burn tests requires sophisticated sampling and analysis systems. Agency personnel responsible for trial burn tests need to be concerned about the accuracy of POHC measurements. Accuracy of such measurements may be assessed by conducting a performance audit. A performance audit is a quantitative assessment of the accuracy of a measurement system. For volatile POHC, a performance audit consists of providing an "unknown" or "blind" cylinder gas to the organization being audited. The organization draws a sample from the cylinder gas through (VOST) or into (bag measurement) the POHC Sampling System and analyzes the collected sample. When a performance audit is conducted during or prior to a hazardous waste trial burn test, it provides an assessment of the measurement accuracy and indicates the presence of any bias for the combined sampling and analysis system.

The EPA's Environmental Monitoring Systems Laboratory operates a program to develop organic gas audit materials and provide these audit gases to federal, state and local agencies or their contractors for use in performance audits during hazardous waste trial burn tests. The Research Triangle Institute (RTI), under contract to the EPA, is the principal organization for development of audit gases and coordinating subsequent audits using these gases. Currently five, six, seven, and nine component gas mixtures have been de-

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Table I. Auditing POHC measurements.[a]

Group I compounds	Group II compounds	Group III compounds	Group IV compounds
Carbon tetrachloride	Trichloroethylene	Vinylidene chloride	Acrylonitrile
Chloroform	1,2-dichloroethane	1,1,2-trichloro-1,2,2-trifluoroethane (F-113)	1,3-Butadiene
Perchloroethylene	1,2-dibromoethane	1,2-dichloro-1,1,2,2,-tetrafluoroethane (F-114)	Ethylene oxide
Vinyl chloride	Acetonitrile	Acetone	Methylene chloride
Benzene	Trichlorofluoromethane (F-11)	1-4 Dioxane	Propylene oxide
	Dichlorodifluoromethane (F-12)	Chlorobenzene	Ortho-xylene
	Bromomethane		
	Methyl ethyl ketone		
	1,1,1-trichloroethane		
Group I Ranges	*Group II Ranges*	*Group III Ranges*	*Group IV Ranges*
7 to 90 ppb	7 to 90 ppb	7 to 90 ppb	7 to 90 ppb
90 to 430 ppb	90 to 430 ppb	90 to 430 ppb	430 to 10,000 ppb
430 to 10,000 ppb			

[a] All gas standards are in a balance gas of nitrogen.

veloped and are available for audits. A list of the gaseous volatile organic compounds and their concentrations are shown in Table I.

The gaseous organic compounds in Table I are purchased in compressed gas cylinders from commercial suppliers. These cylinders, along with an appropriate delivery system, are used directly without dilution in the performance audits. The compressed gas cylinders are especially suitable as an audit material for a number of reasons including simplicity, portability, stability, blind mode of operation (i.e., the user cannot readily ascertain the concentration levels), and ruggedness for interstate shipping.

Before being used as an audit standard, the contents of each cylinder undergoes a series of analyses by the RTI using gas standard prepared and analyzed by the National Bureau of Standards. The NBS gas standards are cylinder gases at the same nominal concentrations as the audit cylinder gases being analyzed. Each audit cylinders gas content is analyzed after initial receipt from the manufacturer to check the accuracy of the reported concentration of each compound. The cylinder contents are then analyzed periodically over the course of a year and later on an annual basis to estimate the compound stability. All concentrations are measured by using gas chromatography (GC) with flame ionization (FID) and/or electron capture detector (ECD). The analytical system and analysis procedures used to determine the compound stability were described previously.[1,2] Emphasis here will be on the logic used in selecting organic compounds to develop audit materials and how these audit materials are successfully used in performance audits to assess and document the accuracy of POHC measurements. Examples of performance audit results are also described in this paper.

Criteria Used for Selection of Organic Compounds

In 1983, EPA initiated a program to develop ppb cylinder gas standards containing hazardous organic compounds that could be used to audit volatile POHC measurement systems during trial burn tests. Through 1985, three mixtures of ppb cylinder gases were developed which are shown in Table I. The criteria used by EPA for selecting various organic compounds in the three mixtures (Groups I, II and III) are described in a separate publication[3]. Briefly, on the basis of experience of EPA's Air and Energy Engineering Research Laboratory (AEERL) with RCRA trial burn testing, five, nine, and seven compound gas standards were developed during 1983 to 1985. Several compounds were included in the same cylinder with the idea that the same gas cylinders could be used to audit more than one POHC during trial burn tests.

In 1986, a six-compound Group IV gas standard was developed. The selection of six organic compounds for Group IV was based on EPA health risk assessment studies plus input from a state agency. The compounds included in the Group IV are also shown in Table I.

In 1987, after considerable interest was shown in the low ppb gas standards by the ambient air measurement community, development of an 18-compound Group V gas standard was initiated (see Table II). The compounds in this gas standard include 16 of the 27 compounds that are presently in Groups I, II, III, and IV gas standards plus two new compounds, ethyl benezene and, 1,2-dichloropropane. The Group V gas standard is intended for auditing ambient air measurement systems and not RCRA trial burn tests.

Since there is an increasing ambient air measurement activity at hazardous waste land fill sites under the Superfund program, a 23 compound—Group VI gas standard is under development during 1988 (see Table III). The compounds in this gas standard include 17 of the 18 compounds that are presently in the Group V gas standards plus six additional compounds. The Group VI gas standard is intended for auditing ambient air measurements at hazardous waste landfill sites. In summary, EPA has developed or has under development, six groups of gas standards for auditing measurement systems during RCRA trial burn tests and/or ambient air measurement programs.

Selection of Compound Concentrations for Audit Standards

The gas standards were initially developed at two concentration ranges to audit the two prevalent test methods for volatile POHCs. The 7–90 ppb and

Table II. Auditing ambient air measurements (Group V).[a]

Carbon tetrachloride	Methylene chloride
Chloroform	Trichlorofluoromethane (F-11)
Perchloroethylene	Bromomethane
Vinyl chloride	Toluene
Benzene	Chlorobenzene
Trichloroethylene	1,3-Butadiene
1,2-dichloroethane	Ortho-xylene
1,2-dibromoethane	Ethyl benzene
1,1,1-trichloroethane	1,2-dichloropropane

[a] The Group V range is 1 to 40 ppb. The balance gas is nitrogen.

90–430 ppb concentration ranges were selected for the Groups I, II, III, and IV Gas Standards to audit POHC measurements during RCRA trial burn tests; the lower range standard is used to audit VOST measurements, and the higher gas standard is used to audit bag sampling measurements. Later, Group I, and IV standards in the 430 to 10,000 ppb concentration range were added. These standards were added because the RCRA regulation requires a destruction and removal efficiency (DRE) of at least 99.99 percent but does not regulate a maximum emission rate for the POHCs. Therefore, some source testers have received approval to conduct the trial burn test in such a manner that POHC concentrations in stack emissions are about 1000 ppb. This reduces considerably the effect of atmospheric POHC contamination on the VOST measurement system, compared to trial burn test where the POHC stack emissions are in the order of 10 to 50 ppb concentrations.

The Groups V and VI gas standard are intended to audit systems that measure hazardous organic compounds in ambient air. Therefore, these gas standards are at lower concentrations, in the range of 1 to 40 ppb, for each compound.

Results and Discussions

Performance Audits

As stated previously, the EPA (through RTI) supplies cylinder gases containing selected POHCs for audits upon request from the federal, state, or local agencies or their contractors. The contractors must, however, be performing hazardous waste trial burn tests on behalf of EPA or one of these other agencies to qualify for the performance audit. In fact, the VOST method described in the EPA manual SW-846 specifically requires an audit during the trial burn.[4] A performance audit must be conducted simultaneously with the actual planned test. A performance audit prior to a trial burn test to assess the proficiency of the measurement system (including the sampling and analytical personnel) is recommended but not required.

When a request is received for an audit cylinder, the cylinder pressure is measured and the cylinder and a heated glass manifold delivery system (when VOST is used for sampling) are shipped by overland carrier to the audit site. General instructions for conducting the audit are included with the audit materials. The audit results are reported to the agency (EPA, state or local) coordinator requesting the audit. There is no charge to the user except the cost of returning the audit cylinder.

As of January 1988, 210 performance audits have been initiated using ppb audit gases. Of these 210 ppb audits, 89 audits have been initiated to assess the accuracy of measurement methods

Table III. Group VI ppb gas mixture currently under development for auditing measurements at hazardous waste landfill sites.[a]

Carbon tetrachloride	Chlorobenzene
Chloroform	1,2-dichloropropane
Perchloroethylene	Vinylidene chloride
Vinyl chloride	Ortho-xylene
Benzene	Methyl ethyl ketone
Trichloroethylene	1,1,2,2-tetrachloroethane
1,1-dichloroethane	Styrene
1,1,1-trichloroethane	trans-1,2 dichloroethylene
Methylene chloride	trans-1,4 dichloro-2-butene
Bromomethane	1,2-dibromoethane
Toluene	Ethyl benzene
Trichlorofluoromethane (F-11)	

[a] The Group VI range will be 1 to 40 ppb. The balance gas is nitrogen. Group VI gas mixture will be available in the fall of 1988.

Table IV. Summary of audits performed for RCRA trial burns (through January 1988).

Group I–IV compounds	Based on 74 completed audits[a]				Frequency VOST auditee results within ±50 percent of audit concentration[e]
	Frequency compounds selected for audits	Measurement system audited			
		VOST[b]	BAG[c]	Other[d]	
Carbon tetrachloride	32	25	4	3	21 out of 25
Chloroform	19	14	2	3	11 out of 14
Perchloroethylene	16	15	1	0	11 out of 15
Vinyl chloride	11	11	0	0	7 out of 11
Benzene	12	12	0	0	9 out of 12
Trichloroethylene	9	8	1	0	7 out of 8
1,2-Dichloroethane	3	3	0	0	3 out of 3
1,2-Dibromoethane	2	2	0	0	1 out of 2
Acetonitrile	3	2	1	0	0 out of 2
Trichlorofluoromethane (F-11)	4	3	0	1	3 out of 3
Dichlorodifluoromethane (F-12)	4	3	0	1	1 out of 3
Bromomethane	3	3	0	0	1 out of 3
Methyl ethyl ketone	2	2	0	0	1 out of 2
1,1,1-Trichloroethane	4	4	0	0	3 out of 4
Vinylidene chloride	2	2	0	0	1 out of 2
1,1,2-Trichloro-1,2,2-Trifluroethane (F-113)	2	2	0	0	2 out of 2
1,2-Dichloro-1,1,2,2-Tetrafluoroethane (F-114)	1	1	0	0	0 out of 1
Acetone	1	1	0	0	1 out of 1
1,4-Dioxane	1	1	0	0	0 out of 1
Toluene	7	6	1	0	4 out of 6
Chlorobenzene	4	4	0	0	4 out of 4
Acrylonitrile	1	0	1	0	0 out of 0
1,3-Butadiene	0	0	0	0	0 out of 0
Ethylene oxide	0	0	0	0	0 out of 0
Methylene chloride	0	0	0	0	0 out of 0
Propylene chloride	0	0	0	0	0 out of 0
Ortho-xylene	0	0	0	0	0 out of 0

[a] Eighty nine audits initiated; 53 audits completed with results; 15 audits currently underway, and 21 audits completed without results.
[b] GC/MS was used for all VOST analysis.
[c] GC with a specific detector (FID/ECD) was used for bag samples.
[d] "Other" includes direct injection and glass bulbs.
[e] Frequency of auditee results within ±50 percent of the audit concentration are 10 out of 11 for bag sampling and six out of eight for "other" methods.

(VOST or bag for sampling and GC/MS or GC with specific detector for analysis) during or prior to hazardous waste trial burn tests. Of the 89 RCRA audits, 74 are complete and the results are summarized in Table IV. For the 74 completed audits, the frequency each compound was selected for audits is shown. Seven of the 27 compounds have been found to be more popular for performance audits. These seven compounds are: carbon tetrachloride, chloroform, perchloroethylene, vinyl chloride, trichloroethylene, benzene, and toluene. Of the 89 audits initiated, 68 are on the VOST, 16 with Tedlar bags, one with glass bulbs and four by direct injections. The majority of the audits employed GC/MS for analysis and only a few used GC with a specific detector. It is very interesting to note from the table that the auditee results for the VOST method are usually within the ±50 percent accuracy limit stated in the VOST protocol.[4]

Currently the VOST method has been validated by EPA for carbon tetrachloride, chloroform, perchloroethylene, benzene, and trichlorofluoromethane (F-11).[5] The high frequency of VOST audit results within the ±50 percent accuracy limit suggests that this method may be an effective means of measuring a large number of POHCs in addition to the five compounds validated.

For the seven month period from July 1987 through January 1988, 28 RCRA trial burn audits were initiated. Of those initiated, 11 audits are complete and the results summarized in Table V. From the results in the table, VOST for sampling and GC/MS for analysis are the methods of choice. As stated previously the most recent audit results are generally within ±50 percent of the audit concentration.

The principal results of these audits have been to assure that analyses are performed properly, to detect problems that could be corrected, and to document the POHC measurement accuracy during hazardous waste trial burn tests.

Stability Studies

To ensure that the concentration of each gas standard has not changed, each standard is periodically analyzed for stability. A two-tier stability assessment is conducted. In the first tier, on each standard, the commercial gas manufacturer conducts a minimum of two analyses that are separated by at least 30 days. The second tier stability assessment involves the periodic reanalysis by RTI. Once a gas standard is received from the commercial gas manufacturer, it is analyzed as soon as possible, then reanalyzed at 2, 6, and 12 months and annually thereafter to determine any change in the gas mixtures. The stability data obtained to date for all the ppb level organics are summarized and published in a separate report.[2] An example of stability data for a Group I ppb gas standard is shown in Table VI. An examination of the stability data for many of the organics in the ppb level cylinder gases show the results varied by less than 10 percent over a one to four year period. A gas standard is not available for auditing purposes until the 2-month RTI analysis is completed. If the compounds are not stable during that period, those compounds are not recommended for audits. It appears from the long term stability study, that all the organics tested (with the exception of ethylene oxide and propylene oxide at the 10-ppb level) are stable enough to be used as reliable audit materials.

Table V. Performance audit results for RCRA trial burn tests.

Audit No.	Sampling system audited	Analytical system audited	Audit compounds	Audit conc (ppb)	Auditee percent bias[a]
199/200	VOST prior to trial burn	GC/MS	Carbon tetrachloride	14.4 ± 0.4	−5
			Toluene	10.2 ± 0.7	−12
			Chlorobenzene	29.8 ± 5.0	−29
188	VOST prior to trial burn	GC/MS	Carbon tetrachloride	37.2 ± 1.1	−13
			Chloroform	8.1 ± 0.2	−22
			Perchloroethylene	9.0 ± 0.2	−11
			Benzene	39.1 ± 1.3	−27
			Vinyl chloride	50.3 ± 1.6	−44
185/186	VOST during trial burn	GC/MS	Trichloroethylene	10.1 ± 0.5	+14
			Toluene	10.2 ± 0.7	+3
181	VOST prior to trial burn	GC/MS	Carbon tetrachloride	291 ± 8.6	−74
			Chloroform	123 ± 2.6	−61
			Perchloroethylene	104 ± 2.6	−52
			Benzene	367 ± 11.8	−84
			Vinyl chloride	416 ± 13.6	−32
180	VOST prior to trial burn	GC/MS	Chlorobenzene	28.3 ± 4.8	−7
170/171	Bag during trial burn	GC/ECD	Acetonitrile	119 ± 8.3	+13
			Acrylonitrile	29.3 ± 2.0	+199
160	VOST during trial burn	GC/MS	Carbon tetrachloride	21.4 ± 2.4	+12
			Chloroform	10.6 ± 0.6	+19
			Perchloroethylene	20.6 ± 1.0	−5
			Benzene	10.3 ± 0.5	+59
			Vinyl chloride	29.4 ± 2.5	−46
159	VOST during trial burn	GC/MS	Carbon tetrachloride	291 ± 8.7	−21
			Chloroform	232 ± 4.9	−25
			Perchloroethylene	365 ± 9.0	−28
			Benzene	300 ± 9.7	+23

[a] Auditee percent bias = $100 \times \frac{\text{Auditee measured conc.} - \text{Audit conc.}}{\text{Audit conc.}}$.

Summary and Recommendations

Compressed gas cylinders containing 27 gaseous volatile organic compounds at parts-per-billion levels have been used sucessfully in audits to assess the accuracy of measurement systems, especially those used during the hazardous waste trial burn tests. The 27 gaseous volatile organic compounds were selected on the basis of the anticipated needs of U.S. EPA's Office of Solid Waste and Office of Air Quality Planning and Standards.

Stability studies indicate that all of the organic compounds tested (with the exception of ethylene oxide and propylene oxide at the 10-ppb level) are stable enough to be used as reliable audit materials. Eighty nine performance audits have been initiated (through January 1988) to assess the accuracy of POHC measurements systems used during hazardous waste trial burn tests. Audit results have generally been within ±50 percent of the RTI measured concentrations.

It is recommended that a performance audit using organic gas cylinders be conducted during each hazardous waste trial burn test as a routine quality assurance procedure. Any federal, state, and local agencies and their contractors planning hazardous waste

Table VI. Example of stability study (Group I compounds).

Activity	Compound[a]				
	Carbon tetrachloride	Chloroform	Perchloro-ethylene	Benzene	Vinyl chloride
Manufacturer analysis					
Theoretical	9.50	39.6	9.40	18.9	20.0
1st analysis date	11/17/83	11/17/83	11/17/83	12/06/83	12/06/83
	7.70	42.1	9.20	15.8	18.3
2nd analysis date	12/16/83	12/16/83	12/16/83	12/15/83	12/15/83
	7.10	41.2	7.70	19.6	19.2
RTI analysis					
1st RTI analysis date	1/06/84	1/06/84	1/06/84	1/23/84	1/23/84
	9.30 ± 0.3	40.0 ± 0.8	9.40 ± 0.2	19.6 ± 0.6	20.2 ± 0.6
2nd RTI analysis day[b]	59	59	59	49	49
	9.30 ± 0.3	38.7 ± 0.8	10.3 ± 0.2	20.0 ± 0.6	19.0 ± 0.6
3rd RTI analysis day	238	238	238	226	226
	9.50 ± 0.3	40.6 ± 0.9	9.50 ± 0.2	19.4 ± 0.6	20.6 ± 0.7
4th RTI analysis day	404	404	404	375	375
	9.60 ± 0.3	37.1 ± 0.8	9.10 ± 0.2	19.4 ± 0.6	20.4 ± 0.7
5th RTI analysis day	on audit	on audit	on audit	on audit	on audit
6th RTI analysis day	833	833	833	810	810
	10.5 ± 0.3	40.5 ± 0.9	11.2 ± 0.3	19.7 ± 0.6	19.4 ± 0.6
7th RTI analysis day	1337	1337	1337	1337	1337
	9.45 ± 0.3	40.6 ± 0.9	10.1 ± 0.3	19.2 ± 0.6	20.1 ± 0.6

[a] Concentrations in ppb.
[b] Number of days after 1st RTI analysis date.

trial burn tests may request a performance audit by contacting Mr. Robert L. Lampe, U.S. EPA, Environmental Monitoring Systems Laboratory, Quality Assurance Division, Research Triangle Park, NC 27711.

Acknowledgments

This project was conducted by the Research Triangle Institute, Research Triangle Park, North Carolina, under contract Numbers 68-02-3767 and 68-02-4125 of the Quality Assurance Division, Environmental Monitoring Systems Laboratory of the U.S. Environmental Protection Agency. Mention of trade names or commercial products does not constitute endorsement or recommendation for use. The paper has been reviewed in accordance with the U.S. Environmental Protection Agency's peer review and administrative review policies and approved for presentation and publication.

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This paper was submitted for peer review February 2, 1988; the revised manuscript was received March 24, 1988.